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PATENT
13DV-13228

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicant: Robert W. Bruce et al.

Serial No.: 09/624,810

Filed: July 24, 2000

Examiner: Rudy Zervigon

Art Unit: 1763

For: ELECTRON BEAM PHYSICAL VAPOR DEPOSITION APPARATUS

DECLARATION UNDER 37 CFR §1.132

Assistant Commissioner for Patents
Washington, D.C. 20231

I, ROBERT W. BRUCE, depose and say that:

(1) I am a co-inventor of the subject matter covered by each of the claims pending in the above-identified U.S. Patent Application Serial No. 09/624,810 ("the Application"), as well as the claims of copending and co-assigned U.S. Patent Application Serial Nos. 09/621,755 (Docket No. 13DV-13220), 09/624,809 (Docket No. 13DV-13041), and 09/624,808 (Docket No. 13DV-13225).

(2) I have been continuously employed with the General Electric Company, GE Aircraft Engines, since 1990, currently as a Senior Staff Engineer, and during the course of my employment have been engaged in the research and development of thermal barrier coating systems for components of gas turbine engines, including the development of equipment and methods for depositing such coating systems.

(3) Claims 1-10 of the above-identified US Patent Application are rejected under 35 USC §102(b) based on a public use or sale of the invention, disclosed in an Information Disclosure Statement filed April 5, 2002.

(4) **Brief Summary of the Claimed Invention:**

I and my co-inventors of the subject matter covered by each of the claims of the Application, conceived and completed, in this country, our disclosed and claimed invention for an electron beam (EB) pattern generated by an electron beam physical vapor deposition (EBPVD) coater to deposit a ceramic thermal barrier coating (TBC) on components (e.g., turbine blades) of a gas turbine engine. Independent claims 1 and 7 require that the coater comprises a coating chamber at an elevated temperature and a subatmospheric pressure, a crucible within the coating chamber, a coating material surrounded by and contained within the crucible and having a surface exposed by the crucible; and an electron beam gun projecting an electron beam onto the surface of the coating material. The gun manipulates the electron beam to generate an EB pattern having a higher intensity at an interface between the surface of the coating material and the crucible than at a central region of the surface of the coating material. A preferred aspect of the invention is that the electron beam is also intentionally projected onto the surface of the crucible itself, and that the EB pattern has a higher intensity on the surface of the crucible than at the central region of the surface of the coating material.

(5) **Summary of the Experimental Use of the EBPVD Coater**

The implementation of the EBPVD coater disclosed in the Application was accomplished in what I believe was an unprecedented twenty months. Furthermore, to my knowledge, the coater is the first and only EBPVD TBC coater that was not completely assembled and tested at the vendor's site (Leybold Systems Gmbh (Leybold), of Hanau, Germany) before installation and test at the purchaser's site (Praxair Surface Technologies, Inc. (Praxair), on behalf of the General Electric Company (GE)). Instead, the components of the coater were assembled and installed at Praxair's facility prior to any system testing. An ambitious schedule was laid out to minimize the time to production, overlapping as many design and assembly steps as possible.

In view of the above, only a bare minimum of tests was performed before initiating what were intended to be, and later confirmed through analysis to be, production coating runs. All tests performed on the EBPVD coater were conducted under my control and nearly always under my personal supervision because, in addition to being deeply involved in the design, assembly, testing and acceptance of the coater, I was also responsible for getting the coater quickly and reliably through its tests to permit the start of production. First there was the independent development and test of the electron beam pattern (pertinent to U.S. Patent Application Serial No. 09/624,810) in April 1998. Then there were independent tests of the heating and coating process parameters (pertinent to U.S. Patent Application Serial Nos. 09/624,809 and 09/621,755), including changes in process parameters (e.g., temperature, pressure) as well as internal process dimensions and the interaction of both. Each of the tests performed to evaluate the EB pattern and process temperature and pressure was done with "button" and other nonhardware specimens, because there are no non-destructive tests for analyzing EBPVD TBC coatings.

Starting in July 1998 and continuing into August 1998, some scrap parts

(blades) were used to further develop heating and coating parameters. The scrap parts were sectioned to verify coating structure and coating thickness distribution over the airfoil and platform of the blades. From these tests, some "good" scrap blades - those with minor dimensional defects but with good bond coat - were used to determine furnace cycle test (FCT) life. FCT life is a well known and critical test performed to evaluate the spallation life of a TBC, which is determined in part by the coating process. Verification of FCT Life took about six weeks, after which the coater was deemed to be ready for the required Vendor Substantiation Evaluation (VSE) initiated on July 23, 1998, during which a few production-quality parts were coated along with scrap parts. A first series of short coating test runs for the VSE was completed on or before July 29, 1998.

At the time of the completion of these initial VSE coating runs, it could not be said whether (a) the production-quality parts processed during VSE were acceptable for installation on a gas turbine engine, or (b) the components of the coater that (i) are subject to degradation during operation of the coater or (ii) effect the acceptability of the coated parts, were beyond the probability of failure. Following VSE the scrap parts and a few of the production-quality parts (at a cost of $4500 each) were put though various destructive tests. Without these destructive tests, there was no way of knowing whether the remaining production-quality parts were acceptable. Because of costs, we did not coat an abundance of production-quality parts during the first VSE coating campaign, but instead coated a representative number of production-quality parts. These parts were loaded through each of the four loading chambers of the coater in three separate coating runs during the VSE. Some of these runs were made at the beginning of a coating campaign, when the process temperature is relatively cool. We then evaporated a lot of ceramic material to build-up the temperature within the coating chamber, permitting us to perform some coating runs to evaluate the coating process on parts in a "hot" coating chamber. This occasion was the first time

the coater attained the "hot" temperature specifically called for as part of the coater acceptance testing required by GE in addition to the VSE. In effect, we were doing portions of the GE acceptance testing and process development in parallel with the VSE in order to accelerate the development time.

So by the end of the VSE, we had coated a bare minimum of production-quality parts, and the coater had been "hot" only once. Because no serious problems were encountered during the VSE, we started what was hoped to be a low volume production run on August 3, 1998, with one shift operated by Praxair personnel (some of whom were new), but again under my control and supervision. However, the acceptability of the very first production-quality parts processed by the coater during the initial VSE runs and the first and subsequent "production" runs was still at issue during an engineering meeting on August 7, 1998, and it was not until September 9, 1998, that these parts (a total of about thirty-six parts) were finally released to production, meaning that each of a number of separate engineering groups responsible for the parts had reviewed and accepted the parts, the last approval occurring on September 9, 1998.

Acceptance of production parts did not signal the completion of testing of the coater or parts processed by the coater. FAA requirements stipulate that parts from each production coating campaign (thirty-five hours of continuous operation) must be metallurgically examined during the first six months of production. Furthermore, multiple VSE coating runs are required by the FAA, typically performed not more frequently than every three months. We accelerated the qualification of the coater by performing a VSE coating run every six weeks on additional part types. Gradually we went to a two-shift and then a three-shift operation. It was not until about twelve months after the initial VSE runs that engineering had sufficient confidence to put the coater into full production.

(6) **Summary of the Experimental Use of Coater Components:**

Crucible: U.S. Patent Application Serial No. 09/624,808

Normal life of a crucible used in an EBPVD coater is about six to nine months. Crucibles are cleaned and visually inspected (but not disassembled) during a "turn-around" cleaning process performed on the coater after each campaign. It takes months if not years before it is apparent that an EBPVD crucible has even a slight problem. A major problem might be spotted as a fast deterioration of the inside edge of the crucible, but that could also be caused by a careless moment of one of the coater operators, and the difficulty is determining whether it is the process or the operator at fault. In any event, the crucible could not have been determined to work for its intended purpose beyond the probability of failure - from deterioration as a result of contacting molten ceramic material and, of significant interest, from the new EB pattern that projects an electron beam directly onto the crucible surface (U.S. Patent Application Serial No. 09/624,810) - for several months after the completion of the VSE.

EB Gun Pressure Protection: U.S. Patent Application Serial No. 09/621,755

The EBPVD coating chamber was designed to extend the life of the electron beam (EB) guns under the high coating process pressure (in excess of 0.010 millibar), which was higher than we had ever before continuously run an EBPVD coater. Normal EB gun life is 100 to 200 hours of continuous operation at lower process pressures (below 0.005 millibar), at which time the gun is disassembled and inspected for damage, etc. With the one-shift coater operation that existed at this phase of the coater testing (i.e., VSE and initial production runs), EB gun life could not have been assessable until after at least two weeks of operation following the start of the VSE. However, according to engineering standards, an EB gun must exhibit acceptable life

seven times in a row in order to be qualified as a success. However, there were mistakes (with potentially catastrophic results) made by new personnel when refurbishing the EB guns of the coater, which negated the gun inspections for several of the coating runs. As a result, at least a couple months passed after VSE before we were satisfied that the coating chamber worked for its intended purpose beyond the probability of failure - protecting the EB guns at elevated process pressures.

EB Pattern: U.S. Patent Application Serial No. 09/624,810

The EB pattern was part of the crucible equation. If an EB pattern is no good it can chew up a crucible, and this particularly EB pattern being utilized with the crucible is quite different from what we previously had experience with. We had tried to get EB guns like those we knew, but to no avail. So the success of the EB pattern was to be measured by the life of the crucible (U.S. Patent Application Serial No. 09/624,808) as well as the number of "spit defects" on coated parts (as discussed in the Application). Molten coating material "spits" that result in spit defects are easily generated by inexperienced or inattentive operators. Because of the new personnel operating the coater, it again took months following VSE before we were able to conclude that the pattern worked for its intended purpose beyond the probability of failure, which was determined by evaluating the damage to the crucible that could be attributed to the EB pattern and by the incidence of spit defects over a statistically significant number of coated parts. Notably, conclusions regarding spitting defects attributable to the EB pattern could not be made with only the parts coated on or before August 4, 1998, because identical parts had been coated up until that time and spit rates and spit rejection rates vary with part configuration as a result of differences in coating procedures and part quality requirements.

Temperature Control: U.S. Patent Application Serial No. 09/624,809

The new coater had to cope with a much greater heat load than what we were familiar with from our existing coater at a GE facility in Cincinnati, Ohio, which necessitated bigger and more effective temperature control features. The existing coater had only one loading chamber, one heating chamber, and one coating chamber. A batch of parts is loaded, then heated, then coated, then unloaded. The twenty-minute coating cycle is interrupted by twenty minutes of unloading, loading and heating. In contrast, the new coater has four loading chambers and two heating chambers, which results in the coating chamber being continuously active (heated). Our experience using the existing coater was with 3000 mm ingots evaporated till part temperatures reached the maximum allowed by the part alloy. The new coater was evaluated on the same basis, while controlled experiments were performed to evaluate different schemes for controlling process temperature. However, the VSE involve only one extended coating campaign, and therefore the effectiveness of the temperature control scheme was not significantly challenged until later, when coating campaigns of at least seventy-two hours were performed. Within a couple of months after VSE, we fixed the procedure of when to move the plate and when to clean around the crucibles to better control process temperature, as disclosed in the Application. Thereafter, we were able to gradually increase the amount of ingot from 3000 mm to 3500 mm and so on until the 7500 mm ingot feed being used now - a process that took about two years following VSE.

(7) I believe that the statements and facts presented above serve to clarify two issues: (a) the extent of the experimental use of the coater and its components; and (b) the supervision and control during this experimental use. Regarding the first issue, I believe that the facts presented above evidence that the initial coating test runs performed on or before August 4, 1998, were not capable of establishing that the

inventions covered by the claims in U.S. Patent Application Serial Nos. 09/621,755, 09/624,808, 09/624,809 and 09/624,810 exhibited practical utility for their intended purposes, and certainly not beyond the probability of failure - the potential for failure loomed over the operation for weeks if not months after completion of the initial VSE runs. As to the issue of "supervision and control," I hereby confirm that I and/or one of my co-inventors was personally present during the tests performed on and before August 4, 1998, and that we - and not Praxair personnel - designed, organized, supervised and controlled those tests.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under §1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Robert W. Bruce

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